Insulating Biomaterials
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Disorders and Stroke

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Introduction

Generally, we are beginning to catch up on compilation of data from the many measurements that have been in progress over the past years and putting the finishing touches on the large electrometer system and animal test instrumentation. We are developing sets of experiments to further define the silicone-silicon dioxide adhesion and encapsulation system. The integrated circuit test devices are still under development. We have also placed emphasis on use of Kapton interconnects for relatively short term implants since it is relatively available and there really is nothing at present to replace it. Also, since silicone plasma depositions are showing good progress, this is becoming a main focus of the chemical engineering collaboration. PPECVD wire coatings and substrate coatings are being pursued.

Instrumentation

One more bank of electrometers were assembled, de-bugged and calibrated this quarter, and is now in use on an 80°C hot block. Temperature controllers were assembled that hold the temperatures to within 1 degree were finished and installed. Ambient temperature and humidity monitoring was implemented. Adapters were constructed to allow testing of sub-dural triple track implants.

New Test Devices

Because of the observed failures of some devices or some portions of encapsulation silicones on some devices which apparently had not been fully cured, cure schedule was investigated this quarter. A simple stretch test was devised using 2mm diameter rods of the materials to be tested. The rods were fabricated by injecting CF20-2186 silicone into 20cm long, 2mm ID Teflon tubes. In order to minimize any possibility of bubble formation, the material was room temperature cured for one day prior to final cure. Four final cure temperatures were used: 50°C, 100°C, 150°C, and 200°C, all for three hours.

Each rod was tied into a loop using a fisherman's knot. Each loop was then folded and tied loosely with a single overhand knot to allow immersion in the bottom of a 1" diameter test tube. One sample of each cure temperature was placed in de-ionized water and maintained at room temperature (~26°C), 80°C, and 90°C. Control samples were maintained at room temperature in air.

Periodically, samples were retrieved and pull tested. Pull test data was normalized to 6cm for the length of the sample when looped over the pull hooks and pre-loaded to 10g. Prior to setting the 10g pre-load, the pull tester was cycled to 60g force and left for 1 minute. Pull testing consisted of stepping 1mm increments followed by measuring the force on the pull hook to 0.1mg resolution. Averaging was used to further improve accuracy. Two cycles were run for each test device, each measurement. Prior to immersion in water, the samples were measured. The first wet measurements were taken 5 days after immersion.

26°C Soak Pull Test of 200°C Cured Nusil CF20-2186

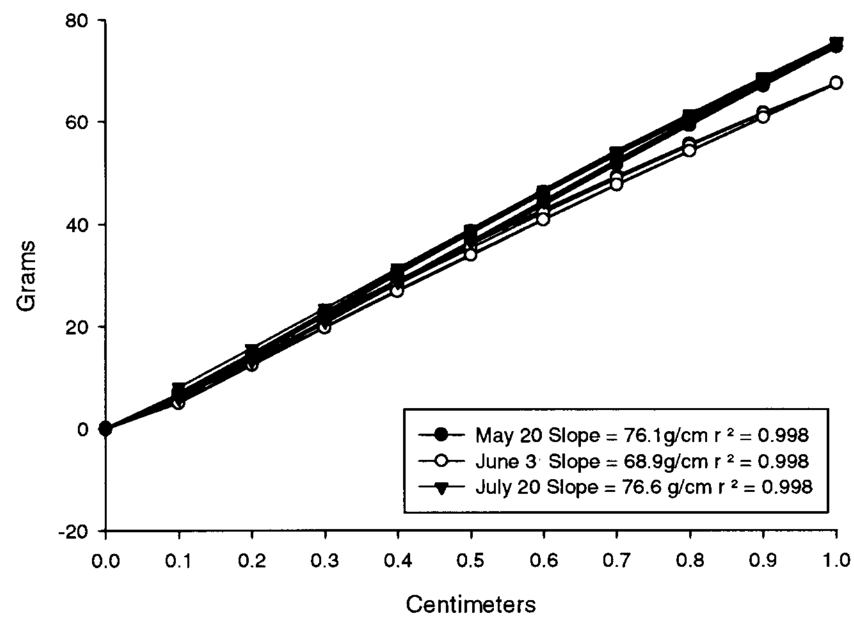

Figure 1: Pull test summary of room temperature deionized water soak of CF20-2186 silicone loop.

80°C Soak Pull Test of 200°C Cured Nusil CF20-2186

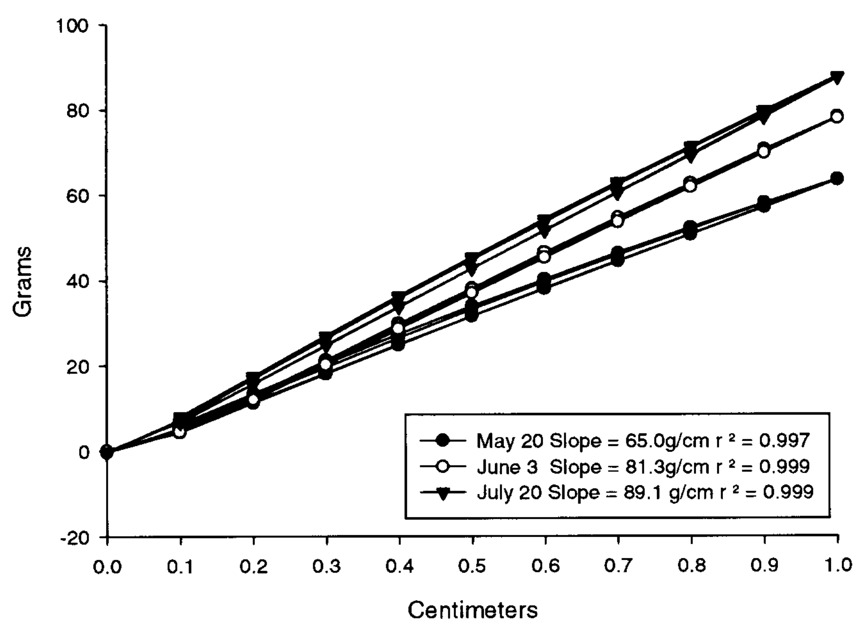

Figure 2: Displacement vs force curves for a silicone loop of Nusil CF20-2186 pre-loaded to 10g. This sample was cured 3 hours at 200C and soaked in 80C de-ionized water for 5 days prior to the first wet measurement.

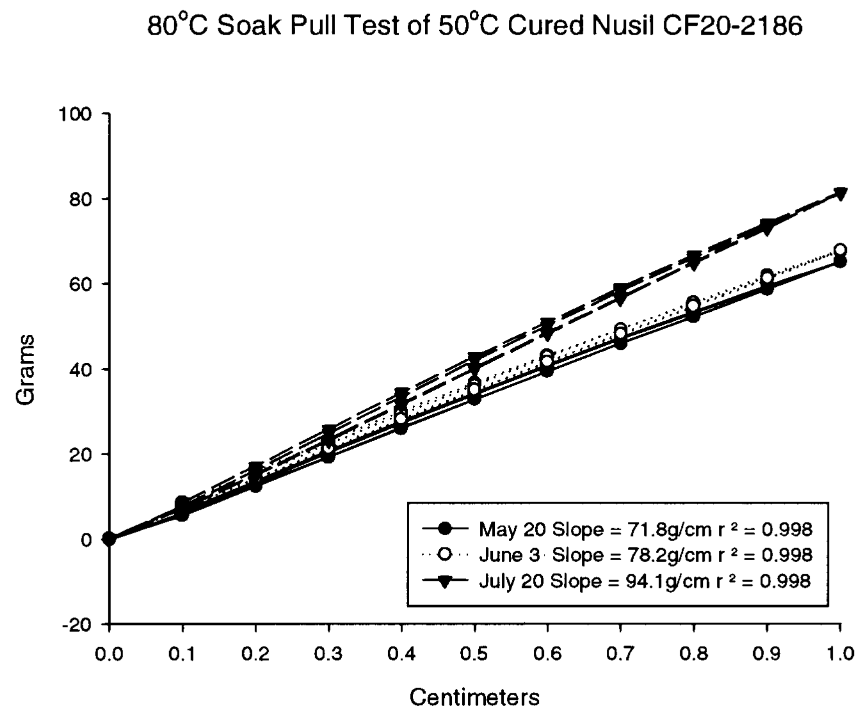

Figure 3: Pull test results of Nusil CF20-2186 sample cured at 50C and soaked at 80C in deionized water.

Figure 1 shows results for Nusil CF20-2186 cured at 200C and soaked at room temperature in deionized water. The odd curve may have been an error during measurement since the first and third measurements agreed so closely. Not much change is evident in this sample.

Figure 2 shows displacement vs force plots for a sample cured at 200C for 3 hours prior to soak. While only a few months have passed, there is a clear shift in the slope of the curves indicating that the sample is possibly cross linking further under the influence of the hot water.

Figure 3 shows results of a sample cured at 50C and soaked at 80C in deionized water. The results indicate a more moderate stiffening of the material than that observed for the 200C cured sample. This may be due to proportionally more depolymerization occuring than in the 200C sample.

A set of these test loops will be fabricated and implanted in animals to further evaluate silicone degradation as a function of cure temperature for promising materials.

New University of Michigan triple track devices were assembled and placed under test. An HF etch was used to ensure a clean surface free of possible metal contamination from the laser ablation of metal defects. This improved the dry resistance readings somewhat but did not substantially improve the readings following saline immersion. It still appears that there is some type of substrate connection in view of the somewhat odd appearing data.

Animal Testing

New adapters were constructed for the animal tester and 7 animals with long term subdural wire loops and triple track implants were tested. Results are summarized in Table 1. Hip triple track devices consisted of four interdigitated electrode arrays (IDE arrays) mounted on titanium percutaneous connectors. Of many that were implanted, only two continue to survive for many years. These devices have continuous 6 volt battery voltage applied between the traces of the IDE arrays. Wire loop implants consisted of 1cm long loops of wire implanted subdurally and leading through the skull to a percutaneous connector. Head triple track devices consisted of 2 IDE arrays implanted subdurally with lead wires through the skull to a percutaneous plug. Channels 8 and 10 were not functional in these implants since there were only 2 IDE arrays.

It is impressive to us that the silicone encapsulated triple tracks have survived for up to 3 years at this point. The silicones used correspond to the animal ID. Implant R4211B was coated with Nusil MED-4211. Implant R2186B was coated with Nusil CF20-2186. Implants R4220B,C were coated with Nusil MED-4220. Implant R2500C was coated with Nusil CV2500, a low volatiles/ultrapure silicone. The R4211B and R2186B implants together have only exhibited one of 8 failures over the 3 year period. The readings indicate a surface resistivity of between 1E16 to 1E18 Ω/sq. The newer head implant are exhibiting similar performance. The Teflon coated wire loops are also performing in similar fashion to the wire loop tests run in-vitro.

	Channel >		4	6	8	10		
Animal ID	Type	Years	Interdigitated Electrode Ohms				Date Tested	Date Implanted
R4211B	Hip TT	3.0	1.96E+14	3.35E+12	1.36E+11	1.98E+12	07/09/98	07/11/95
R2186B	Hip TT	2.8	<1 E+9	>2 E+14	>2 E+14	>2 E+14	07/09/98	10/03/95
R3PITA	Wire Loops	1.1	4.91E+13	2.27E+13	1.32E+13	1.78E+13	07/09/98	05/20/97
R2PT3A	Wire Loops	1.1	1.96E+14	6.81E+13	5.27E+13	<1 E+9	07/09/98	05/30/97
R4220B	Head TT	0.9	1.35E+11	>2 E+14	NA	NA	07/08/98	08/05/97
R4220C	Head TT	0.9	>2 E+14	2.04E+14	NA	NA	07/08/98	08/07/97
R2500C	Head TT	0.8	9.82E+12	<1 E+9	NA	NA	07/08/98	10/06/97

Table 1: Summary of surviving subcutaneous triple track implants over the hip (Hip TT) sub-dural wire loop implants (Wire Loops), and sub-dural triple track implants (Head TT) in rabbits.

Vapor Phase Silicone Depositions

Because of the promising results obtained with silicone depositions thus far, a small set of test samples was constructed. Using processing and plasma conditions detailed in Progress Report 6, a set of samples were prepared using three power conditions and two monomers (D3 and D4). Films of each type were deposited onto four clean silicon squares which were then assembled into soak tubes. Silicone O-rings (0.25" dia) were glued to the surface of the films using Nusil CF20-2186 silicone. Wires were attached to the backside of the samples using conductive epoxy. All but the center opening through the O-ring was coated with CF20-2186. Teflon insulated leadwires exited the long glass testing tube through a Teflon/silicone seal. **Table 2** summarizes the deposition conditions, measured capacitance between the silicon substrate and the saline solution, thickness, and deposition rate. Thickness and deposition rates were estimated from the capacitance, estimated area, and estimated relative dielectric constant (3). Sample RSPLL04 may have been thinner than anticipated due to an error in estimation of the total deposition time (sample gas ran out).

Sample ID	Monomer	On/Off	Dep(min)	Cap(pF)	~Thick(ang)	Rate
RSPLL07	D3	10/100	38	708	11870	312
RSPLL03	D4	10/100	60	483	17399	290
RSPLL06	D3	100/400	60	274	30671	511
RSPLL04	D4	100/400	50	732	11481	230
RSPLL05	D3	Continuous	60	193	43544	726

Table 2: Summary of plasma depositions of 2 monomers and three plasma conditions.

During assembly of the samples into the testing tubes, the continuous samples wrinkled up following application and curing of the O-ring. The cause of this was not clear. Only the area outside the O-ring wrinkled. Inside the O-ring was still intact, smooth, and non-wrinkled. The D4 continuous sample was not fully assembled in order to allow assessment of this phenomenon.

Once the testing tubes were assembled, saline spiked with <0.01% silver was added to immerse the test devices in the bottom of the tube. A platinum reference wire was used to provide drive voltage for the IV sweep. The testing tubes were placed in a temperature regulated hot block well and held at 80°C throughout testing. Voltage applied to the saline was swept from -5 to +5 in one and 0.1 volt steps (near zero) in order to expand the dynamic range of the automated system described in prior progress reports. The range of the system was approximately 1e10-1e15 ohms. Samples were tested "continuously", but resistance was computed at the end of each IV sweep which requires about 4 days at present. Results are shown in Figures 1-5. The two low on/off ratio depositions (10/100) are shown in figures 1 and 2. These samples show good resistance with only a slight downward trend over time after the first few days under soak. One sample in each failed near the beginning of testing probably from particulate contamination of the substrate (by microscopical observations). (Failures can be detected in the graphs by observing the absence of points - randomly bubbles perhaps plug the defects for a given reading). Figures 3 and 4 show results from the 100/400 on/off depositions. In these samples, about half have defects which may have been due to film cracking. Figure 5 shows results from a continuous deposition. Only one of the four samples yielded good results over time. The other three films had developed localized micro-cracks possibly from contamination or residual film stress.

This preliminary test data indicates that plasma deposited silicones may be useful materials for coating of devices for long term implantation in biological systems. Detailed analysis of the chemical structure of the films is in progress, and additional test depositions of the more promising monomers under a variety of pulsed conditions will be accomplished. Suitability of the films as wire coatings will also be investigated. Once the films are more developed, triple track devices will be prepared, coated and tested in saline and implanted in animals.

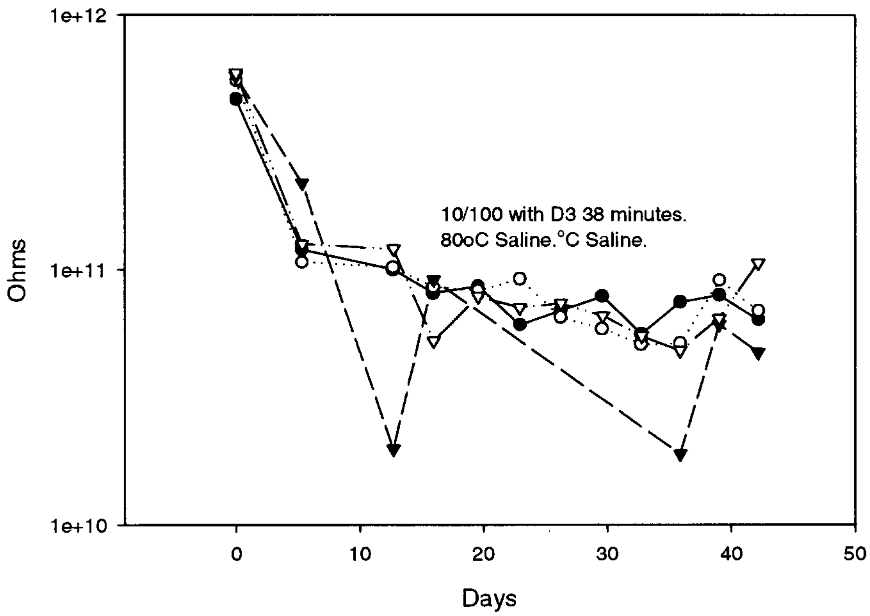

Figure 4: Sample RSPLL7 on/off 10/100 with D3 monomer for 38 minutes. 80°C saline soak.

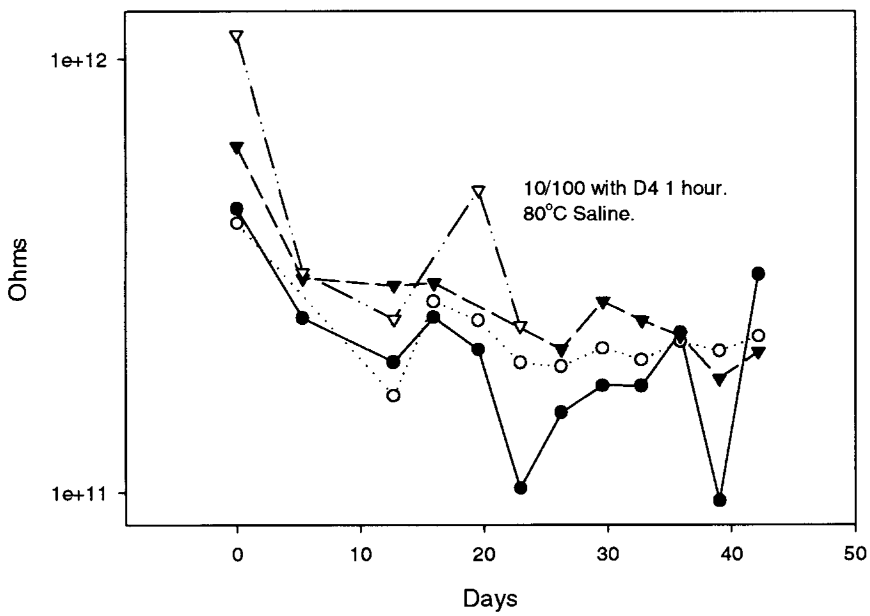

Figure 5: Sample RSPLL3 on/off 10/100 with D4 monomer for 60 minutes. 80°C saline soak.

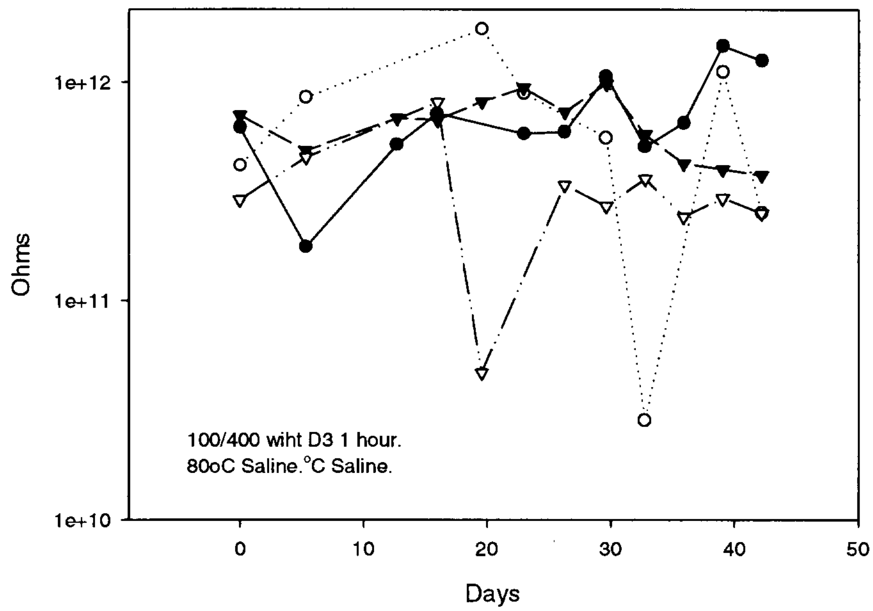

Figure 6: Sample RSPLL6 on/off 100/400 with D3 monomer for 60 minutes. 80°C saline soak.

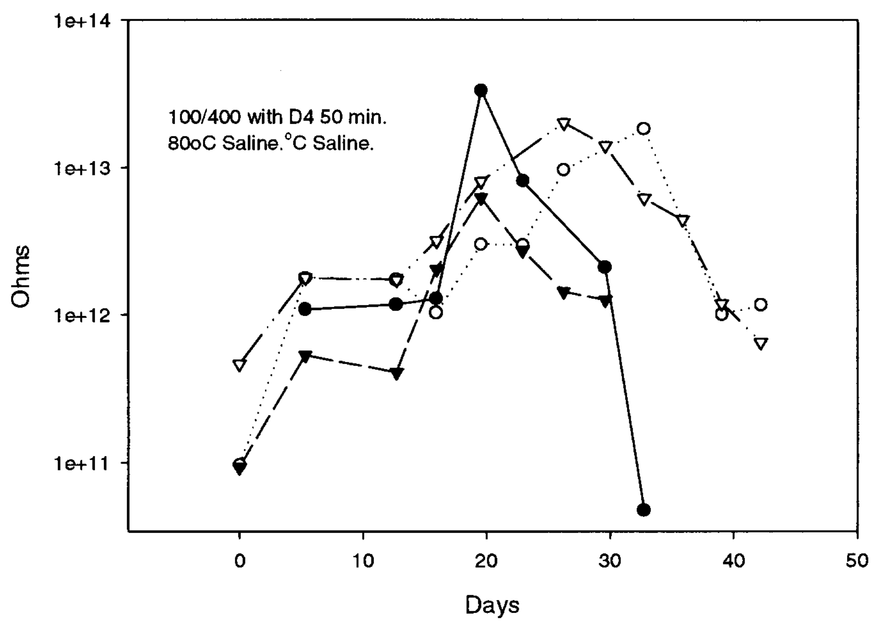

Figure 7: Sample RSPLL4 on/off 100/400 with D4 monomer for 50 minutes. 80°C saline soak.

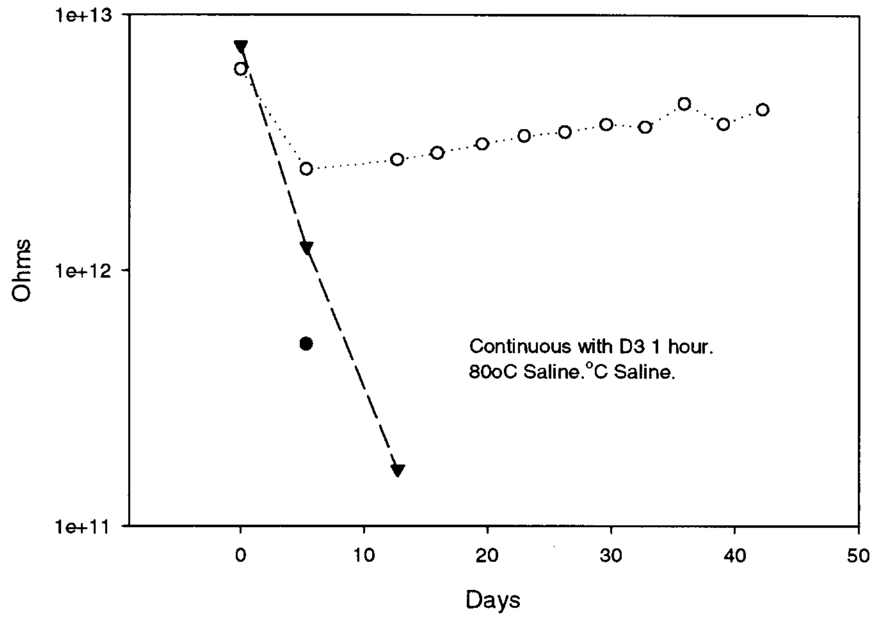

Figure 8: Sample RSPLL5 on/off continuous with D3 monomer for 60 minutes. 80°C saline soak.

Integrated Circuit Test Chips

The integrated circuit test chips were received from MOSIS and evaluated. During the final phase of the design, a subcircuit was replaced with an updated subcircuit but one necessary connection was not re-established. Unfortunately this error happened to occur in the current source used to down-integrate the output capacitor. Without an output it was impossible to operate the chip. However, the basic amplifier stages, shift register stages, and pulsed output stages were operational on a circuit development chip giving confidence that a re-submission would be successful. Thus the circuit was corrected, and additional details were double checked to improve odds for success during the next fabrication run. The revised circuit will be available for testing in September.

Kapton Micro-Ribbon Cable Encapsulation: One of the pressing needs for an insulating biomaterial is a substrate for micro-ribbon interconnects that can be used to interconnect University of Michigan silicon microelectrode arrays with percutaneous connectors. Silicon cables are fragile and at present may be unable to withstand the forces generated by healing tissues such as dura, skull, and periosteum. A more robust cable is needed to interconnect a skull mounted percutaneous connector and a sub-durally implanted silicon microelectrode array with integral micro-ribbon.

Polyimide based micro-ribbon cables are being developed for providing interconnection between the silicon micro-ribbon/microelectrode array and the skull mounted percutaneous connector. Polyimide is known to be a poor insulating biomaterial for long-term implants because of relatively rapid degradation in aqueous environments. However, polyimide is one of the few flexible substrate materials for which there is an established micro-fabrication technology. Mylar is another choice, but Mylar also exhibits similar long term deficiency.

For short-term implants, perhaps up to one year, polyimide may be sufficiently long lived to provide an interim solution. Over the past year, we have been testing Kapton based interdigitated test structures insulated with various materials. A recent data summary is shown in Table 3. Only devices that were completely encapsulated in one of several epoxies showed promise. All acrylic and epoxy sandwich structures failed rapidly in water soak tests due to diffusion of water into residual spaces left during the assembly process. Also, all silicone insulated structures failed rapidly.

The University of Michigan National Center for Neural Communication Technology (Jamie Hetke) has fabricated several test devices with a commercially produced mini-ribbon cable that used standard Kapton sandwich structures with acrylic adhesives. Several of these assemblies were sent to us for testing, and all failed rapidly by similar mechanisms. An additional note is that the copper traces on all commercially fabricated test ribbons darkened during soak testing indicating that oxidation of the copper traces was taking place in spite of the acrylic/Kapton sandwich structure.

Gary Roughton in Dr. Nagle's lab at North Carolina State University prepared five different Kapton based interdigitated electrode arrays to test various approaches to preparation of implantable Kapton based structures. All structures were formed by deposition of gold traces on Kapton substrates followed by coating with an insulator. The insulators used were Dow Corning silicone T-RTV, Dupont Polyimide 2721, Dupont Polyimide 2723, Dupont PC1025, and Dupont Dow3-1753. Figure 9 - Figure 13 summarize results from early soak tests. Initially, samples were dry tested for 3 weeks before the addition of saline to establish a baseline of bulk resistivity. Following addition of saline, many samples survived for an additional 2 weeks. Failures can be noted by graphs where data points are missing because the amplifiers saturated from the high current levels. The Dupont Polyimide 2721 coated samples were the most reliable thus far, with all four devices surviving saline soak for over 14 days. This performance is far better than that previously achieved using commercially prepared Kapton sandwich structures, and they are still under test. This information was passed along to both the Nagle lab and the University of Michigan.

Sample ID	Ribbon	Encap Class	Encap	1st Saline	Initial Wet	Date	Reading	Days	Units
KTT27A	Kapton/Acrylic	Epoxy	Epotek 377	01/01/98		03/05/98	2.60E-11	63	Amps@10v
KTT23B	Kapton/Acrylic	Epoxy	Epotek 353	01/01/98		02/13/98	4.00E-09	43	Amps@10v
KTT17A	Bare Kapton	Epoxy	Epotek 377	05/13/97	3.00E-13	02/13/98	5.00E-09	276	Amps@10v
KTT1GA	Bare Kapton	Epoxy	Epotek 87-GT	06/02/97	5.00E-12	09/17/97	1.00E-08	107	Amps@10v
KTT25F	Kapton/Acrylic	Acrylic Epoxy	Epotek 715	09/02/97	8.00E-12	02/13/98	1.70E-08	164	Amps@10v
KTT13A	Bare Kapton	Epoxy	Epotek 353	05/13/97	1.20E-13	02/13/98	3.00E-08	276	Amps@10v
KTT25CB	Kapton/Acrylic	Acrylic Epoxy	Epotek 716	06/13/97	1.30E-08	02/24/98	3.00E-08	256	Amps@10v
KTT25A	Kapton/Acrylic	Acrylic Epoxy	Epotek 715	06/04/97	2.00E-09	02/13/98	4.50E-08	254	Amps@10v
KTT25D	Kapton/Acrylic	Acrylic Epoxy	Epotek 715	07/15/97	7.90E-11	03/05/98	8.00E-08	233	Amps@10v
KTT25CA	Kapton/Acrylic	Acrylic Epoxy	Epotek 715	06/11/97	9.50E-08	02/26/98	1.20E-07	260	Amps@10v
KTT25E	Kapton/Acrylic	Acrylic Epoxy	Epotek 715	07/15/97	6.50E-10	02/24/98	4.00E-06	224	Amps@10v
KTT1PA	Bare Kapton	Polyimide		01/01/98		03/03/98	5.00E-06	61	Amps@10v
KTT16A34	Bare Kapton	Silicone	CF20-2186	03/16/97	3.61E+04	05/25/97	failed	70	Ohms
KTT16A56	Bare Kapton	Silicone	CF20-2186	03/16/97	8.15E+07	05/25/97	failed	70	Ohms
KTT16A78	Bare Kapton	Silicone	CF20-2186	03/16/97	5.29E+07	05/25/97	failed	70	Ohms
KTT16A910	Bare Kapton	Silicone	CF20-2186	03/16/97	2.49E+07	05/25/97	failed	70	Ohms
KTT1PB	Bare Kapton	Polyimide			Faulty Asm		failed	0	Amps@10v
KTT23A	Kapton/Acrylic	Epoxy	Epotek 353		Faulty Asm		failed	0	Amps@10v
KTT26A34	Kapton/Acrylic	Silicone	CF20-2186	03/17/97	5.51E+07	04/06/97	failed	20	Ohms
KTT26A56	Kapton/Acrylic	Silicone	CF20-2186	03/17/97	1.26E+08	05/20/97	failed	64	Ohms
KTT26A78	Kapton/Acrylic	Silicone	CF20-2186	03/17/97	3.17E+07	04/06/97	failed	20	Ohms
KTT26A910	Kapton/Acrylic	Silicone	CF20-2186	03/17/97	7.80E+02	04/06/97	failed	20	Ohms
KTT28A	Kapton/Acrylic	Acr Adhesive	3M DP805	05/01/97	1.00E-04	06/14/97	failed	44	Amps@10v
KTT2GA	Kapton/Acrylic	Epoxy	Epotek 87-GT	06/10/97	2.70E-08	06/12/97	failed	2	Amps@10v
KTT3BA34	Kap/Acr Sand	None	None	03/23/97	5.56E+09	05/26/97	failed	64	Ohms
KTT3BA56	Kap/Acr Sand	None	None	03/23/97	8.40E+09	05/26/97	failed	64	Ohms
KTT3BA78	Kap/Acr Sand	None	None	03/23/97	8.46E+09	05/26/97	failed	64	Ohms
KTT3BA910	Kap/Acr Sand	None	None	03/23/97	4.58E+09	05/26/97	failed	64	Ohms
KTT43A	Kapton/Epoxy	Epoxy	Epotek 353	05/16/97	1.70E-08	06/07/97	failed	22	Amps@10v
KTT47A	Kapton/Epoxy	Epoxy	Epotek 377	05/16/97	2.90E-08	05/29/97	failed	13	Amps@10v
KTT5BA34	Kap/Epo Sand	None	None	05/12/97	1.75E+06	05/28/97	failed	16	Ohms
KTT5BA56	Kap/Epo Sand	None	None	05/12/97	1.73E+06	05/28/97	failed	16	Ohms
KTT5BA78	Kap/Epo Sand	None	None	05/12/97	2.62E+05	05/28/97	failed	16	Ohms
KTT5BA910	Kap/Epo Sand	None	None	05/12/97	1.59E+06	05/28/97	failed	16	Ohms

Table 3: Results of Kapton Interdigitated Electrode Array testing.

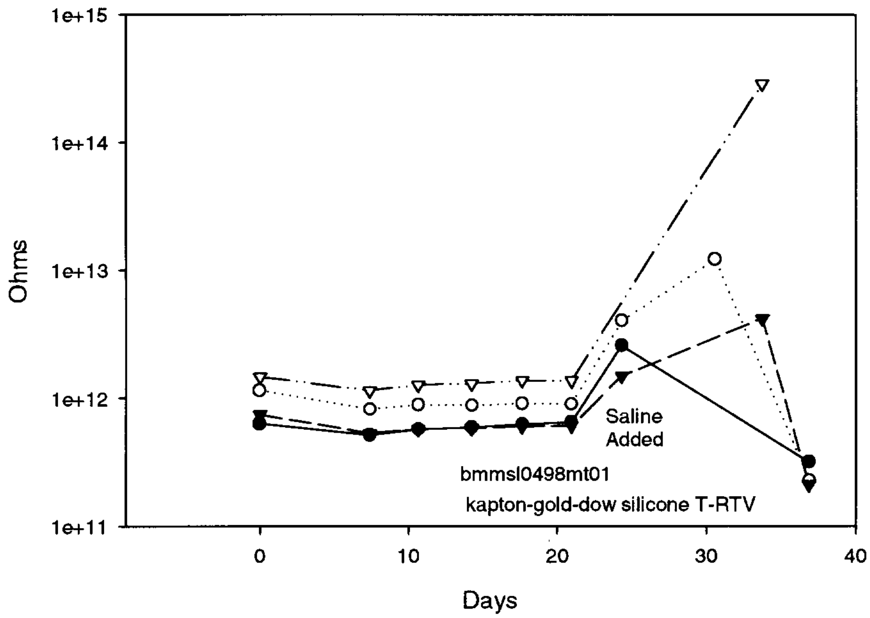

Figure 9: Long term soak data from North Carolina State University Kapton interdigitated electrode array test structure with gold traces and insulated with Dow Corning Silicone T-RTV.

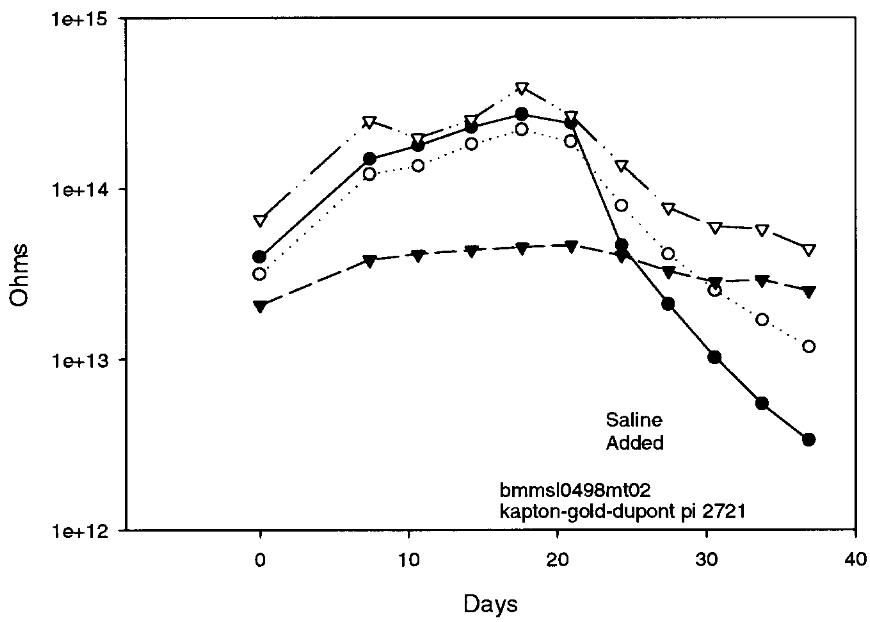

Figure 10: Long term soak data from North Carolina State University Kapton interdigitated electrode array test structure with gold traces and insulated with Dupont Polyimide-2721.

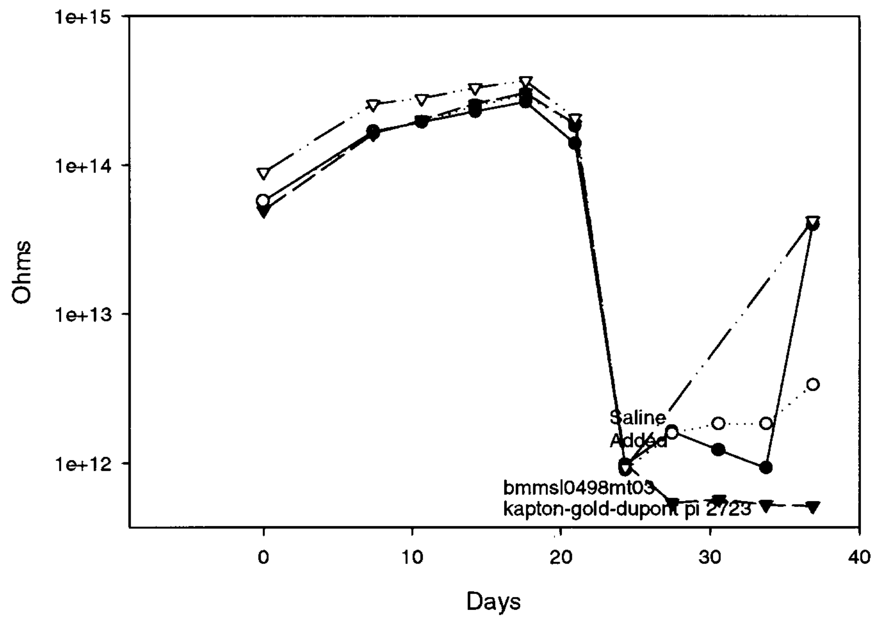

Figure 11: Long term soak data from North Carolina State University Kapton interdigitated electrode array test structure with gold traces and insulated with Dupont Polyimide-2723.

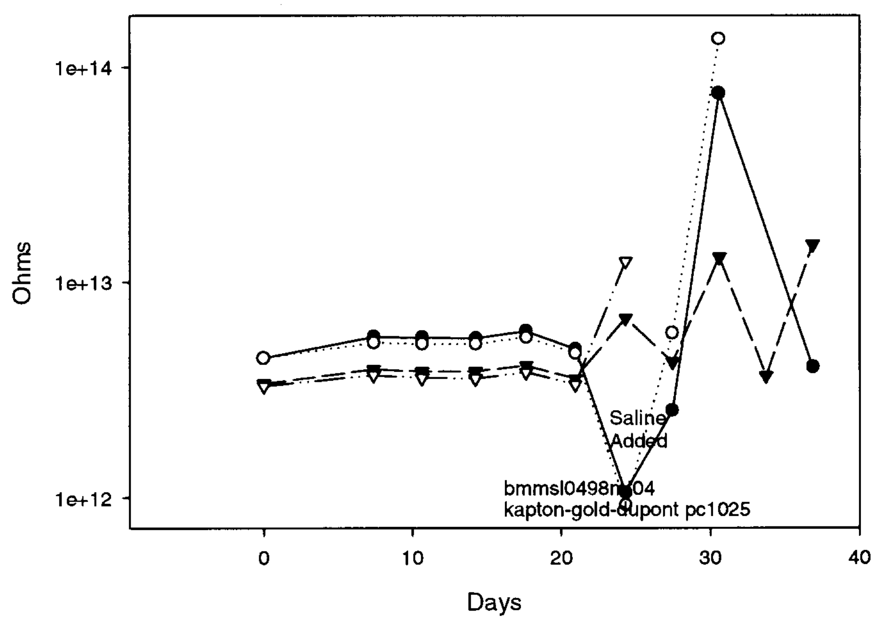

Figure 12: Long term soak data from North Carolina State University Kapton interdigitated electrode array test structure with gold traces and insulated with Dupont PC1025.

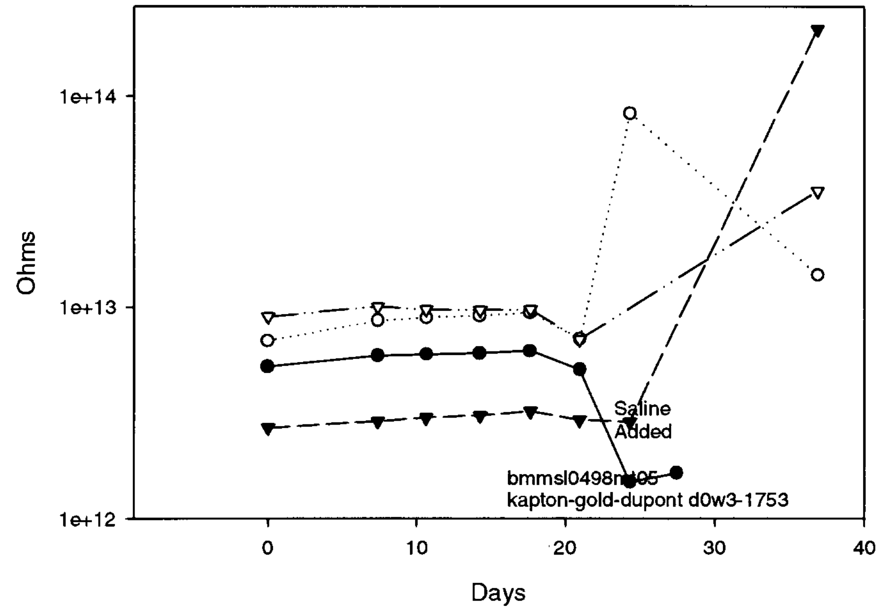

Figure 13: Long term soak data from North Carolina State University Kapton interdigitated electrode array test structure with gold traces and insulated with Dow Corning 3-1753.